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Performance of Europium-Doped Strontium Iodide, Transparent Ceramics and Bismuth-loaded Polymer Scintillators

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ABSTRACT

Recently discovered scintillators for gamma ray spectroscopy, single crystal SrI₂(Eu), GYGAG(Ce) transparent ceramic and Bismuth-loaded plastics, offer resolution and fabrication advantages compared to commercial scintillators, such as NaI(Tl) and standard PVT plastic. Energy resolution at 662 keV of 2.7% is obtained with SrI₂(Eu), while 4.5% is obtained with GYGAG(Ce). A new transparent ceramic scintillator for radiographic imaging systems, GLO(Eu) offers high light yield of 70,000 Photons/MeV, high stopping, and low radiation damage. Implementation of single crystal SrI₂(Eu), Gd-based transparent ceramics, and Biloaded plastic scintillators can advance the state-of-the art in ionizing radiation detection systems.

Keywords: Scintillators, strontium iodide, garnets, bixbyites, transparent ceramics, gamma ray spectrometers, radiography scintillators, plastic scintillators

1. INTRODUCTION

Rapid unambiguous isotope identification is facilitated by detectors offering high energy resolution gamma ray spectroscopy and large active volumes. Deployment of radioisotope identification detectors (RIIDs) offering energy resolution significantly superior to Thallium-doped Sodium Iodide NaI(Tl) is now feasible. The single crystal scintillator, $SrI_2(Eu)$, offers energy resolution for gamma spectroscopy comparable to that of Lanthanum Bromide doped with Cerium, LaBr₃(Ce) and considerably better than Thallium-doped Sodium Iodide, NaI(Tl) [1-3]. Encapsulated 1 in single crystals of Europium-doped Strontium Iodide, $SrI_2(Eu)$, now routinely provide energy resolution at 662 keV of <3%.

Transparent ceramics are fully dense monoliths of micron-scale crystallites, formed by sintering high purity ceramic nanopowders, generally of a single pure phase cubic crystal structure [4-7]. While the best energy resolution for gamma ray spectroscopy is obtained with single crystal scintillators, such as $SrI_2(Eu)$ and $LaBr_3(Ce)$, transparent ceramics offer the possibility of low-cost, net-shape fabrication of oxide scintillators, with benefits of mechanical ruggedness, stability in air and radiation hardness. Transparent ceramic Cerium-doped Gadolinium Garnet, GYGAG(Ce), fabricated at LLNL, offers 4.5% resolution at 662 keV with PMT readout, and with low-noise silicon photodetector readout, <4% can be obtained.

Due to their availability in large sizes at low cost, plastic scintillators, generally based on Polyvinyltoluene, PVT, are used in many applications where a higher density material exhibiting a photopeak would be preferred. Recently, we reported that cm-scale Bismuth-loaded plastic scintillators can provide energy resolution of <9% at 662 keV [3]. Applications such as neutron-interrogation screening, in which a pulsed neutron source irradiates an object of interest and the resultant capture gamma rays are analyzed to identify the materials comprising the object, require large scintillator detectors with modest energy resolution but fast timing. The Bi-loaded plastic is comprised of elements with low neutron activation, should be formable into very large sizes, and offer adequate stopping and energy resolution for effective performance in this application.

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For radiographic imaging applications, we are developing transparent ceramic Europium-doped bixbyites [8]. While pure $Lu_2O_3(Eu)$ forms transparent ceramics with low enough optical scatter for some applications when conditions are optimized, a small amount of secondary phase tends to be present, reducing optical transparency. We therefore have been exploring the more phase-stable mixed Bixbyite, Gadolinium Lutetium Oxide, $(Gd,Lu)_2O_3(Eu)$.

2. EQUIPMENT AND METHODS

 $SrI_2(3 \text{ mol}\%Eu)$ crystals are being grown at RMD, Oak Ridge National Laboratory and Fisk University using the vertical Bridgman method. The crystals are grown in silica ampoules using anhydrous SrI_2 beads (Aldrich, 99.99-99.999%) and EuI_2 powder (Aldrich, 99.9-99.999%), as the starting materials [9-11]. Crystals from all three growers have offered similar performance. Eu-doped crystals are cut into a tapered cylinder shape, while undoped SrI_2 crystals are used as right cylinders, polished and encapsulated in an aluminum cans at LLNL [12].

Transparent ceramic garnets were formed at LLNL using stoichiometric mixed metal oxide particles synthesized via flame spray pyrolysis (FSP), a nanoparticle production method developed by Pratsinis and co-workers [13] and Laine and co-workers [14]. Synthesis of FSP nanoparticles was carried out at LLNL, as well as by Nanocerox, Inc. The FSP nanoparticles are formed into a green body, vacuum sintered, then hot-isostatic pressed into optically transparent parts [8]. The relative ease with which the GYGAG(Ce) ceramic is formed with high transparency is due to the "intersubstitutional" cations, whereby the three different cation sites of the garnet unit cell may be occupied by more than one of the cations. Similarly, the phase stability of the other transparent ceramic discussed in this paper, $(Gd_1Lu)_2O_3(Eu)$, is superior to that of $Lu_2O_3(Eu)$, making it readily formable with excellent transparency.

Polymer scintillators with 40wt% Bismuth were formed at LLNL by mixing a Bismuth metallo-organic with monomer and 3% fluor, heating to melt, and then subsequently polymerizing the homogeneous mixture. Two different fluors were investigated, an Iridium complex, emitting in the green, and diphenylanthracene, emitting in the blue.

Beta radioluminescence employed ⁹⁰Sr/⁹⁰Y source (~1 MeV average beta energy). Radioluminescence spectra were collected with a Princeton Instruments/Acton Spec 10 spectrograph coupled to a thermoelectrically cooled CCD camera. The scintillation light produced by the samples under excitation with a ¹³⁷Cs source (662 keV gamma) or with a ²²⁶Ra source was detected by a commercially available Hamamatsu R6231-100 PMT. The signals were shaped with a Tennelec TC 244 spectroscopy amplifier and recorded with an Amptek MCA8000-A multi-channel analyzer for offline analysis. Decay times of scintillator materials were obtained with a Golden Engineering pulsed x-ray source coupled to an oscilloscope. The Scintillator Light Yield Non-proportionality Characterization Instrument, located at LLNL, is based on the Compton Coincidence technique. Details of the instrumentation and use of the data to obtain intrinsic energy resolution estimates may be found in refs. 15 and 16, respectively.

3. RESULTS AND DISCUSSION

3.1 Characteristics of Single Crystal Europium-doped Strontium Iodide

We are working to identify the properties that make $SrI_2(Eu)$ such a high-performance scintillator. One possible origin of its high light yield of ~80,000 Photons/MeV is a relatively small bandgap, together with the low phonon frequencies characteristic of iodides that minimize energy loss to phonons during the relaxation of high energy carriers during the cascade. Figure 1 shows the absorption and radioluminescence spectra measured using a 1 mm thick undoped Strontium Iodide crystal. Strong absorbance at 310 nm, with a tail to lower energy, indicates a bandgap of ~4 eV. The radioluminescence spectrum is comprised of a small band centered at 400 nm, presumably due to the SrI_2 self-trapped exciton, and a "long-wave" band extending through the visible, with peak value at 550 nm, which is likely due to impurity- or defect-mediated emission. Also shown in Figure 1 is the radioluminescence spectrum of a typical Europium-doped Strontium Iodide crystal, dominated by the Eu^{2+} emission band at 420 nm, along with a small amount of the "long-wave" band. The relative intensity of the "long-wave" band varies considerably from crystal to crystal, as a function of both the Eu^{2+} doping level and of the crystal purity, and similar effects have been previously reported with other alkaline earth halides, such as $Ericmath{BaI_2(Eu)}$ [9,17]. In Figure 2, a pulse height spectrum acquired with a $Ericmath{BaI_3(Eu)}$ source using a 13 cm $Ericmath{BaI_2(Eu)}$ shows resolution of 2.7% at 662 keV (shaping time 12 $Ericmath{BaI_3(Eu)}$). This performance is comparable to that of Cerium-doped Lanthanum Bromide, with the advantage that $ErIcmath{BaI_2(Eu)}$ is free of intrinsic radioactivity.

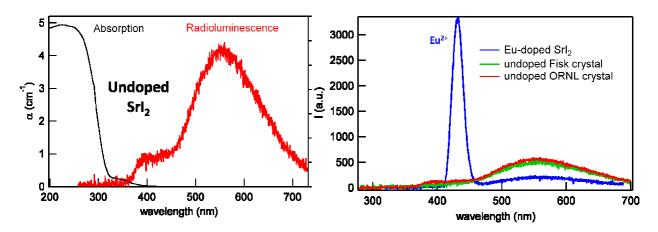


Figure 1. (left) Absorption and beta-excited radioluminescence spectra of and undoped Strontium Iodide crystal. (right) Radioluminescence spectra of Eu-doped and undoped SrI_2 crystals. Note that the self-trapped exciton band lies to slightly higher energy than the Eu^{2+} emission, explaining the excellent scintillation efficiency of $SrI_2(Eu)$.

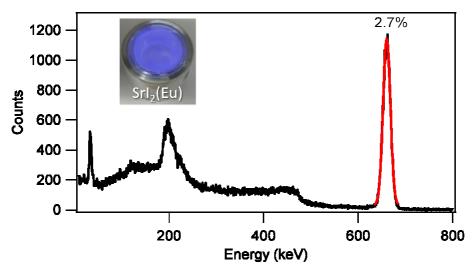


Figure 2. Pulse height spectrum acquired with a 137 Cs source using an encapsulated 13 cm 3 SrI $_2$ (3%Eu) crystal, offering 2.7% resolution at 662 keV. (inset) Photograph of a 13 cm 3 encapsulated SrI $_2$ (Eu) crystal under UV illumination.

3.2 Characteristics of Gadolinium Garnet Ceramic Scintillator

Gadolinium Yttrium Gallium Aluminum Garnet (GYGAG) transparent ceramics are being developed at LLNL for gamma ray spectroscopy applications [5-7, 18]. Reported values for the bandgaps of Yttrium Aluminum Garnet (YAG) and of Gadolinium Gallium Garnet (GGG) are 6.7 eV [19] and 4.6 eV [20], respectively. The absorption spectra of undoped and Ce-doped GYGAG are shown in Figure 3. Several salient features include the sharp Gd³⁺ absorption lines that lie within the bandgap, and the absorption onset at 230 nm corresponding to a band gap of ~5.4 eV, in good agreement with the bandgap for the mixed garnet GYGAG lying about halfway between those of the line compounds YAG and GGG. Also shown in Figure 3 is the scintillation decay profile for GYGAG(Ce), acquired using pulsed x-ray excitation. The decay exhibits two principal components: the fast time constant of 250 ns may be assigned to direct

trapping of excitation on Ce^{3+} , and its subsequent emission, while the slower component, at ~1.7 microseconds may be assigned to excitation trapping first at Gd^{3+} and then transferring to Ce^{3+} . The advantage of addition of Gd to the garnets is the augmentation of their light yield and light yield proportionality [21], however, the total decay is somewhat longer than that of the well-known scintillators YAG(Ce) or LuAG(Ce) [see for example ref. 22], due to the mechanism of energy transfer via Gd.

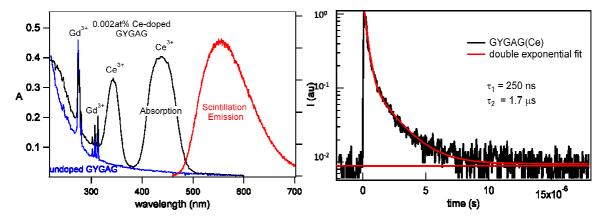


Figure 3. (left) Absorption spectra of two GYGAG ceramics, undoped and Ce-doped, along with the beta-excited radioluminescence spectrum of GYGAG(Ce). (right) The scintillation decay for GYGAG(Ce) acquired with pulsed x-ray excitation shows that the scintillation intensity decreases by 100x from the initial value within $4 \mu s$, the typical shaping time used for this scintillator.

In Figure 4, a pulse height spectrum acquired with a 137 Cs source using a cubic-cm size GYGAG(Ce) shows resolution of 4.5% at 662 keV (shaping time of 4 μ s) with PMT readout. This performance is superior to that of NaI(Tl), which typically offers 6.5% resolution at 662 keV, with the advantage that the oxide transparent ceramic GYGAG(Ce) is mechanically and environmentally robust.

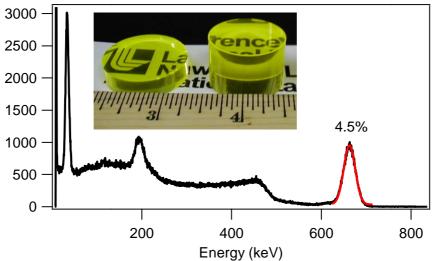


Figure 4. Pulse height spectrum acquired with a ¹³⁷Cs source shows that a 1 cm³ GYGAG(Ce) transparent ceramic scintillator with PMT readout provides 4.5% resolution at 662 keV. (inset) Photograph of a 5.3 cm³ and a 9.9 cm³ GYGAG(Ce) ceramic.

3.3 Comparative Gamma Ray Spectroscopy with SrI₂(Eu), GYGAG(Ce), LaBr₃(Ce), NaI(Tl) and Germanium

To study the effect of energy resolution on the ability of detectors to measure and discriminate weak gamma lines within a background, we performed measurements with a 226 Ra source, shown in Figure 5. The 226 Ra source has a large number of closely-spaced gamma lines. The spectrum acquired with a mechanically cooled semiconductor Germanium detector displays >20 peaks. The energy resolution obtainable with $SrI_2(Eu)$ and $LaBr_3(Ce)$ is adequate to clearly identify about 17 of them—all of the strong lines that are well-separated, as well as several that are weak and closely spaced. The resolution with GYGAG(Ce) is better than with NaI(Tl), as noted by the narrower peaks, as well as a few features it resolves better than NaI(Tl), such as the feature at \sim 400 keV.

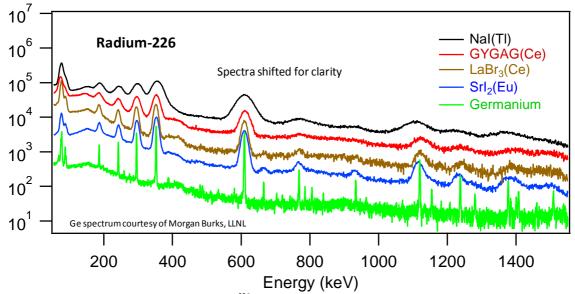


Figure 5. Gamma spectra acquired with a ²²⁶Ra source using a mechanically-cooled Germanium detector, and the following scintillators, SrI₂(Eu), LaBr₃(Ce), GYGAG(Ce) transparent ceramic and NaI(Tl), all with PMT readout, using shaping times adjusted for the best performance with each scintillator.

3.4 Characteristics of Bismuth-loaded Plastic Scintillators for Gamma Ray Spectroscopy

We are developing a class of plastic scintillators containing a high loading of a Bismuth organometallic compound, in order to convey high gamma stopping while not degrading the scintillation light yield. Two samples were characterized for their gamma and electron light yield proportionality, shown in Figure 6. The gamma proportionality measurements utilized the following sources: ²⁴¹Am (59.5 keV), ⁵⁷Co, (122 keV), ¹³³Ba (31.6, 81, 279.5 and 356 keV), ¹³⁷Cs (escape peak at 583 and photopeak at 661.7 keV). The electron non-proportionality was obtained using the Scintillation Light Yield Non-proportionality Characterization Instrument (SLYNCI) [15,16]. The two plastic samples were identical, except that one sample contained 3% Diphenylanthracene, or DPA, while the other contained 3% of an Iridium complex fluor known as FIrpic. The scintillation decay for the DPA-doped sample is 14 ns, while the FIrpic-doped sample decays in about 1.25 µs. It is notable for both the electron and gamma measurement that the sample doped with the Iridium complex exhibits better proportionality. We can estimate that the non-proportionality-limited energy resolution at 662 keV for the DPA- and FIrpic-doped samples is 3.5 and 2.3%, respectively [21]. Therefore, if this performance can be maintained for large sizes, the DPA-doped material could offer sufficient energy resolution with fast enough timing for use in neutron interrogation applications. The FIrpic-doped material offers potential for high energy resolution gamma spectroscopy, due to the capacity for such Iridium complexes to accept excitation from both singlet and triplet excitons. This mechanism is well-known for applications in Organic Light Emitting Diodes, and also

previously demonstrated for scintillation [22, 23]. Therefore, the light yield and proportionality of the FIrpic-doped sample is enhanced compared to the DPA-doped material, in which only singlets may be emitted.

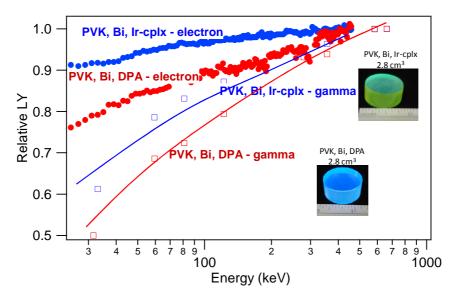


Figure 6. Light yield proportionality obtained with gamma excitation (open symbols and solid lines) and with electron excitation using SLYNCI (closed symbols). The proportionality of the sample using the Iridium complex (spin-orbit couping fluor) is somewhat better than the DPA-doped sample.

3.5 Characteristics of Gadolinium Lutetium Oxide Bixbyite Radiography Scintillator Ceramic

Transparent ceramic Lu₂O₃(Eu) is a known material, though achieving acceptable transparency has been a challenge [9, 24]. We have identified a mixed Bixbyite, (Gd,Lu)₂O₃(5%Eu), that is very phase-stable, thus formable with low optical scatter in sheets or voxels for high energy radiography. GLO(Eu) offers high density of 8-9 g/cm³ and a high light yield of 70,000 Photons/MeV. In Figure 7, the beta excited radioluminescence spectra of standard materials used for scintillator-based radiography CsI(Tl), CdWO₄ are compared with GLO(Eu). Standard non-intrusive inspection systems use a millisecond-pulsed 9 MeV Bremsstrahlung x-ray source (average energy 3 MeV), and readout can be made following a variety of configurations, from lens-based imaging to linear photodiode arrays. The light yield, α_{γ} (the attenuation coefficient of 3 MeV x-rays), Φ_{PD} (the detection sensitivity of Silicon at the emission maximum of the scintillator) are best with GLO(Eu), as shown in Table 1. The radiation hardness of GLO(Eu) was tested by exposure to 450 keV x-rays. No darkening was observed even for the longest exposure, of 10 kGy. Use of GLO(Eu) in non-destructive inspection systems could reduce the dose required to achieve accurate radiographs and permit new geometries to be utilized, not readily available with CsI(Tl) and CdWO₄ single crystals.

| Scintillator | Density (g/cm ³) | α _γ (cm ⁻¹) @ 3MeV | Light yield Ph/MeV | Φ _{PD} @ λ _{max} | Principal decay (µs) | Afterglow (%@ 100ms) | Comments |
|--------------|------------------------------|--|-----------------------|------------------------------------|-------------------------|-------------------------|-----------|
| GLO | 9.1 | 0.36 | 70,000 | 1.0 @611 | 1000 | 0.01 | No issues |
| CsI(Tl) | 4.5 | 0.17 | 65,000 | 0.96 @550 | 8 | 0.3 | Degrades |
| $CdWO_4$ | 7.9 | 0.30 | 28,000 | 0.88 @500 | 5 | 0.02 | Toxic |

Table 1. Properties of several optically transparent scintillators used for high energy radiography.

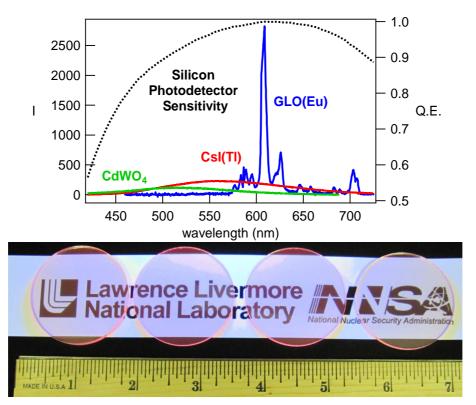


Figure 7. (top) Beta radioluminescence spectra of transparent ceramic GLO(Eu) compared with CdWO₄ and CsI(Tl), spectra may be directly compared and represent their absolute light yields. (bottom) A photograph of four 4.2 cm diameter by 0.2 cm thick GLO(Eu) radiography ceramics in room lights and under weak UV illumination, displaying the orange Eu³⁺ emission.

4. CONCLUSIONS

We have identified several new scintillator materials and are developing them for multiple applications. Among them, the highest light yield, and most proportional material is $SrI_2(Eu)$. Transparent ceramic GYGAG(Ce) offers high light yield, and gamma spectroscopy with better resolution than NaI(Tl). We have synthesized and characterized Biloaded polymer scintillators that can be used for gamma spectroscopy. High light yields and excellent phase stability are obtained for a new radiography scintillator, GLO(Eu). We are now optimizing fabrication procedures and scaling up to larger sizes for all of these materials.

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